

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

5-(1*H*-Indol-3-ylmethylidene)-2,2-dimethyl-1,3-dioxane-4,6-dione

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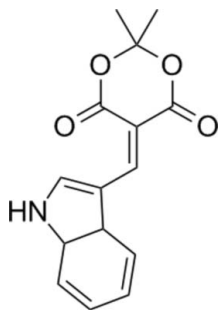
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Received 23 May 2011; accepted 19 June 2011

Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.041; wR factor = 0.099; data-to-parameter ratio = 14.4.

In the title compound, $\text{C}_{15}\text{H}_{13}\text{NO}_4$, the conjugated double-bond system between the two rings adopts a *cis* configuration and there is an intramolecular indole–ketone $\text{C}-\text{H}\cdots\text{O}$ interaction. The indole $\text{N}-\text{H}$ group forms an intermolecular hydrogen bond with a ketone O -atom acceptor, giving a chain structure along the **ab** direction. The O -heterocyclic ring adopts a boat conformation and makes a dihedral angle of 16.72 (6)° with the indole ring system.

Related literature

 For a similar structure, see: He *et al.* (2011).


Experimental

Crystal data

$\text{C}_{15}\text{H}_{13}\text{NO}_4$
 $M_r = 271.26$
 Triclinic, $P\bar{1}$
 $a = 7.0228$ (4) Å
 $b = 8.7021$ (5) Å
 $c = 11.5668$ (9) Å
 $\alpha = 80.281$ (6)°
 $\beta = 76.362$ (6)°
 $\gamma = 70.662$ (5)°
 $V = 645.02$ (7) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 150$ K
 $0.30 \times 0.25 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur Eos CCD-detector diffractometer
 Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
 $T_{\min} = 0.969$, $T_{\max} = 1.000$
 5351 measured reflections
 2632 independent reflections
 2098 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.099$
 $S = 1.04$
 2632 reflections
 183 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O4}^i$	0.88	2.02	2.8285 (16)	152
$\text{C8}-\text{H8}\cdots\text{O3}$	0.95	2.17	2.8492 (19)	128

 Symmetry code: (i) $x + 1, y - 1, z$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

The authors thank Mr Zhi-Hua Mao of Sichuan University for the X-ray data collection. This study was supported by the Research Fund of the Key Laboratory of TCM Biotechnology (Xihua University).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2118).

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 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2011). E67, o1835 [doi:10.1107/S1600536811023944]

5-(1*H*-Indol-3-ylmethylidene)-2,2-dimethyl-1,3-dioxane-4,6-dione

Yu-Xin He, Jin-Wei Wu, Rong-Sheng Tong, Jin-Qi Li and Jian-You Shi

S1. Comment

The title compound C₁₅H₁₃NO₄ (I) is a key reaction intermediate which can be used to synthesize the 4(1*H*)quinolone derivatives *via* thermolysis. These compounds can be used as precursors for the synthesis of anti-malarial and, anticancer agents.

In (I) (Fig. 1) the conjugated double bond system between the two rings adopts a *cis* configuration and there is an intramolecular indole C—H···O_{ketone} interaction. The indole N—H group forms an intermolecular hydrogen bond with a ketone O acceptor (Table 1), giving a one-dimensional chain structure.

S2. Experimental

A mixture of 2,2-dimethyl-1,3-dioxane-4,6-dione (1.44 g, 0.01 mol) and methyl orthoformate (1.27 g, 0.012 mol) was heated to reflux for 0.5 h, after which a solution of 1*H*-indole (1.17 g, 0.01 mol) in ethanol (20 mL) was added. The mixture was refluxed for a further 3.5 h and then poured into cold water after which the product was removed by filtration. Yellow crystals of (I) were obtained after 7 days from the room temperature evaporation of a solution in CH₂Cl₂–methanol.

S3. Refinement

Hydrogen atoms were included in the refinement at calculated positions and allowed to ride on the parent atom with C—H = 0.95, 0.98 Å or N—H = 0.88 Å and $U_{\text{iso}} = 1.2U_{\text{eq}}$ (aromatic C or N) or $1.5U_{\text{eq}}$ (aliphatic).

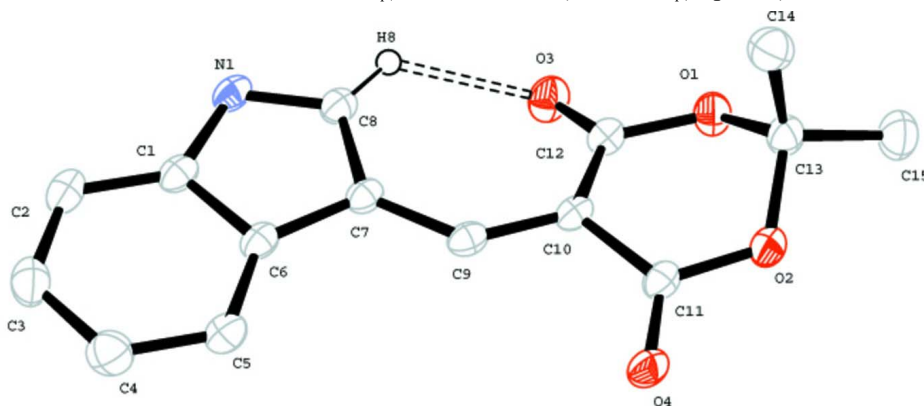


Figure 1

The molecular conformation and atom numbering scheme of the title compound with non-H atoms shown as 50% probability ellipsoids. The intramolecular hydrogen bond is shown as a dashed line.

5-(1*H*-Indol-3-ylmethylidene)-2,2-dimethyl-1,3-dioxane-4,6-dione*Crystal data*

$C_{15}H_{13}NO_4$	$Z = 2$
$M_r = 271.26$	$F(000) = 284$
Triclinic, $P\bar{1}$	$D_x = 1.397 \text{ Mg m}^{-3}$
Hall symbol: $-P\ 1$	Mo $K\alpha$ radiation, $\lambda = 0.7107 \text{ \AA}$
$a = 7.0228 (4) \text{ \AA}$	Cell parameters from 2300 reflections
$b = 8.7021 (5) \text{ \AA}$	$\theta = 2.9\text{--}29.2^\circ$
$c = 11.5668 (9) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$\alpha = 80.281 (6)^\circ$	$T = 150 \text{ K}$
$\beta = 76.362 (6)^\circ$	Block, yellow
$\gamma = 70.662 (5)^\circ$	$0.30 \times 0.25 \times 0.20 \text{ mm}$
$V = 645.02 (7) \text{ \AA}^3$	

Data collection

Oxford Diffraction Xcalibur Eos CCD-detector diffractometer	5351 measured reflections
Radiation source: fine-focus sealed tube	2632 independent reflections
Graphite monochromator	2098 reflections with $I > 2\sigma(I)$
Detector resolution: $16.0874 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.018$
ω scans	$\theta_{\text{max}} = 26.4^\circ$, $\theta_{\text{min}} = 2.9^\circ$
Absorption correction: multi-scan	$h = -8 \rightarrow 8$
(<i>CrysAlis PRO</i> ; Oxford Diffraction, 2010)	$k = -10 \rightarrow 10$
$T_{\text{min}} = 0.969$, $T_{\text{max}} = 1.000$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.099$	$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 0.1554P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
2632 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
183 parameters	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.44545 (15)	0.08780 (12)	0.63114 (9)	0.0267 (3)
O2	0.13409 (15)	0.23138 (12)	0.74782 (9)	0.0258 (3)
O3	0.68858 (15)	-0.09984 (13)	0.71118 (10)	0.0310 (3)

O4	0.06872 (15)	0.18683 (12)	0.94348 (10)	0.0261 (3)
N1	0.79371 (17)	-0.49318 (14)	0.96643 (12)	0.0228 (3)
H1	0.9001	-0.5746	0.9390	0.027*
C1	0.6924 (2)	-0.48713 (17)	1.08487 (14)	0.0219 (3)
C2	0.7324 (2)	-0.59954 (18)	1.18294 (15)	0.0270 (4)
H2	0.8446	-0.6975	1.1762	0.032*
C3	0.6019 (2)	-0.56276 (19)	1.29094 (15)	0.0309 (4)
H3	0.6237	-0.6370	1.3603	0.037*
C4	0.4373 (2)	-0.41693 (19)	1.29963 (15)	0.0308 (4)
H4	0.3488	-0.3950	1.3749	0.037*
C5	0.4011 (2)	-0.30476 (18)	1.20145 (14)	0.0249 (3)
H5	0.2901	-0.2061	1.2088	0.030*
C6	0.5306 (2)	-0.33914 (17)	1.09113 (14)	0.0203 (3)
C7	0.5402 (2)	-0.25491 (17)	0.97153 (14)	0.0201 (3)
C8	0.7072 (2)	-0.35817 (17)	0.89988 (14)	0.0218 (3)
H8	0.7521	-0.3355	0.8165	0.026*
C9	0.3935 (2)	-0.10549 (17)	0.94238 (14)	0.0206 (3)
H9	0.2906	-0.0669	1.0096	0.025*
C10	0.3646 (2)	-0.00301 (17)	0.83978 (14)	0.0205 (3)
C11	0.1806 (2)	0.14000 (17)	0.85038 (14)	0.0216 (3)
C12	0.5113 (2)	-0.01466 (17)	0.72654 (14)	0.0229 (3)
C13	0.2287 (2)	0.16020 (18)	0.63629 (14)	0.0251 (4)
C14	0.1329 (2)	0.0340 (2)	0.62248 (15)	0.0307 (4)
H14B	-0.0138	0.0865	0.6219	0.046*
H14C	0.1492	-0.0518	0.6894	0.046*
H14A	0.2011	-0.0145	0.5472	0.046*
C15	0.2029 (3)	0.3017 (2)	0.54051 (16)	0.0351 (4)
H15A	0.2694	0.2613	0.4622	0.053*
H15C	0.2665	0.3795	0.5551	0.053*
H15B	0.0561	0.3565	0.5418	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0235 (5)	0.0276 (6)	0.0241 (6)	-0.0043 (4)	-0.0026 (4)	0.0005 (5)
O2	0.0264 (6)	0.0207 (5)	0.0253 (6)	-0.0002 (4)	-0.0056 (5)	-0.0019 (4)
O3	0.0216 (6)	0.0307 (6)	0.0320 (7)	-0.0010 (5)	0.0005 (5)	-0.0023 (5)
O4	0.0239 (5)	0.0216 (5)	0.0261 (6)	0.0007 (4)	-0.0010 (5)	-0.0055 (4)
N1	0.0183 (6)	0.0172 (6)	0.0302 (8)	-0.0004 (5)	-0.0044 (5)	-0.0059 (5)
C1	0.0191 (7)	0.0181 (7)	0.0306 (9)	-0.0067 (6)	-0.0061 (6)	-0.0042 (6)
C2	0.0258 (8)	0.0187 (7)	0.0364 (10)	-0.0054 (6)	-0.0096 (7)	-0.0001 (7)
C3	0.0356 (9)	0.0270 (8)	0.0306 (10)	-0.0108 (7)	-0.0107 (7)	0.0046 (7)
C4	0.0310 (8)	0.0323 (9)	0.0277 (9)	-0.0108 (7)	-0.0007 (7)	-0.0037 (7)
C5	0.0227 (8)	0.0211 (7)	0.0290 (9)	-0.0050 (6)	-0.0026 (6)	-0.0042 (6)
C6	0.0181 (7)	0.0168 (7)	0.0284 (9)	-0.0065 (6)	-0.0065 (6)	-0.0036 (6)
C7	0.0185 (7)	0.0174 (7)	0.0258 (8)	-0.0057 (6)	-0.0055 (6)	-0.0041 (6)
C8	0.0204 (7)	0.0198 (7)	0.0258 (8)	-0.0048 (6)	-0.0058 (6)	-0.0041 (6)
C9	0.0181 (7)	0.0188 (7)	0.0263 (8)	-0.0062 (6)	-0.0032 (6)	-0.0064 (6)

C10	0.0177 (7)	0.0164 (7)	0.0261 (8)	-0.0032 (6)	-0.0030 (6)	-0.0047 (6)
C11	0.0212 (7)	0.0170 (7)	0.0270 (9)	-0.0059 (6)	-0.0052 (6)	-0.0024 (6)
C12	0.0219 (7)	0.0197 (7)	0.0263 (9)	-0.0059 (6)	-0.0036 (6)	-0.0029 (6)
C13	0.0231 (8)	0.0246 (8)	0.0233 (9)	-0.0021 (6)	-0.0036 (6)	-0.0025 (6)
C14	0.0300 (9)	0.0319 (9)	0.0304 (10)	-0.0084 (7)	-0.0065 (7)	-0.0048 (7)
C15	0.0391 (9)	0.0302 (9)	0.0303 (10)	-0.0055 (7)	-0.0078 (8)	0.0037 (7)

Geometric parameters (Å, °)

O1—C12	1.3644 (18)	C5—C6	1.395 (2)
O1—C13	1.4326 (17)	C6—C7	1.450 (2)
O2—C11	1.3559 (18)	C7—C8	1.402 (2)
O2—C13	1.4430 (18)	C7—C9	1.4123 (19)
O3—C12	1.2084 (17)	C8—H8	0.9500
O4—C11	1.2165 (18)	C9—H9	0.9500
N1—H1	0.8800	C9—C10	1.372 (2)
N1—C1	1.3877 (19)	C10—C11	1.4649 (19)
N1—C8	1.3359 (19)	C10—C12	1.458 (2)
C1—C2	1.384 (2)	C13—C14	1.511 (2)
C1—C6	1.4063 (19)	C13—C15	1.504 (2)
C2—H2	0.9500	C14—H14B	0.9800
C2—C3	1.380 (2)	C14—H14C	0.9800
C3—H3	0.9500	C14—H14A	0.9800
C3—C4	1.405 (2)	C15—H15A	0.9800
C4—H4	0.9500	C15—H15C	0.9800
C4—C5	1.380 (2)	C15—H15B	0.9800
C5—H5	0.9500		
O1—C12—C10	116.72 (12)	C5—C6—C7	134.43 (13)
O1—C13—O2	109.75 (12)	C6—C5—H5	120.7
O1—C13—C14	110.44 (12)	C7—C8—H8	125.1
O1—C13—C15	106.62 (12)	C7—C9—H9	112.5
O2—C11—C10	117.29 (13)	C8—N1—H1	124.7
O2—C13—C14	110.71 (12)	C8—N1—C1	110.52 (12)
O2—C13—C15	105.59 (12)	C8—C7—C6	105.50 (12)
O3—C12—O1	117.16 (13)	C8—C7—C9	131.37 (15)
O3—C12—C10	125.97 (14)	C9—C7—C6	122.98 (13)
O4—C11—O2	116.98 (12)	C9—C10—C11	116.17 (13)
O4—C11—C10	125.70 (14)	C9—C10—C12	125.57 (13)
N1—C1—C6	107.03 (13)	C10—C9—C7	135.03 (14)
N1—C8—C7	109.82 (14)	C10—C9—H9	112.5
N1—C8—H8	125.1	C11—O2—C13	118.28 (11)
C1—N1—H1	124.7	C12—O1—C13	118.58 (11)
C1—C2—H2	121.5	C12—C10—C11	117.87 (13)
C1—C6—C7	107.12 (13)	C13—C14—H14B	109.5
C2—C1—N1	129.50 (13)	C13—C14—H14C	109.5
C2—C1—C6	123.46 (14)	C13—C14—H14A	109.5
C2—C3—H3	119.6	C13—C15—H15A	109.5

C2—C3—C4	120.86 (15)	C13—C15—H15C	109.5
C3—C2—C1	116.95 (14)	C13—C15—H15B	109.5
C3—C2—H2	121.5	H14B—C14—H14C	109.5
C3—C4—H4	119.2	H14B—C14—H14A	109.5
C4—C3—H3	119.6	H14C—C14—H14A	109.5
C4—C5—H5	120.7	C15—C13—C14	113.52 (14)
C4—C5—C6	118.70 (14)	H15A—C15—H15C	109.5
C5—C4—C3	121.57 (15)	H15A—C15—H15B	109.5
C5—C4—H4	119.2	H15C—C15—H15B	109.5
C5—C6—C1	118.44 (14)		
C13—O1—C12—O3	165.01 (13)	C3—C4—C5—C6	0.7 (2)
C13—O1—C12—C10	-19.01 (18)	C4—C5—C6—C1	0.1 (2)
C12—O1—C13—O2	49.24 (16)	C4—C5—C6—C7	179.37 (16)
C12—O1—C13—C14	-73.09 (16)	C1—C6—C7—C8	0.37 (17)
C12—O1—C13—C15	163.15 (13)	C1—C6—C7—C9	176.32 (14)
C13—O2—C11—O4	-164.73 (13)	C5—C6—C7—C8	-178.93 (17)
C13—O2—C11—C10	17.27 (19)	C5—C6—C7—C9	-3.0 (3)
C11—O2—C13—O1	-48.22 (16)	C6—C7—C8—N1	0.17 (17)
C11—O2—C13—C14	73.94 (16)	C9—C7—C8—N1	-175.30 (16)
C11—O2—C13—C15	-162.80 (14)	C6—C7—C9—C10	-179.25 (17)
C8—N1—C1—C2	-179.28 (16)	C8—C7—C9—C10	-4.5 (3)
C8—N1—C1—C6	0.89 (17)	C7—C9—C10—C11	177.42 (16)
C1—N1—C8—C7	-0.66 (18)	C7—C9—C10—C12	-9.9 (3)
N1—C1—C2—C3	-178.55 (15)	C9—C10—C11—O2	-171.64 (13)
C6—C1—C2—C3	1.3 (2)	C9—C10—C11—O4	10.6 (2)
N1—C1—C6—C5	178.68 (13)	C12—C10—C11—O2	15.1 (2)
N1—C1—C6—C7	-0.75 (17)	C12—C10—C11—O4	-162.73 (15)
C2—C1—C6—C5	-1.2 (2)	C9—C10—C12—O1	173.13 (14)
C2—C1—C6—C7	179.40 (14)	C9—C10—C12—O3	-11.3 (3)
C1—C2—C3—C4	-0.3 (2)	C11—C10—C12—O1	-14.3 (2)
C2—C3—C4—C5	-0.7 (2)	C11—C10—C12—O3	161.31 (15)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O4 ⁱ	0.88	2.02	2.8285 (16)	152
C8—H8 \cdots O3	0.95	2.17	2.8492 (19)	128
C9—H9 \cdots O4	0.95	2.37	2.7979 (18)	107

Symmetry code: (i) $x+1, y-1, z$.